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REINFORCEMENT OF GUAYULE RUBBER WITH NON-OIL DEPENDENT FILLERS, APPLICATIONS IN TANK TRACK PADS

APRIL, 1982

CONTRACT NO. DAAK30-79-C-0108

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The Guayule Rubber, a non-petroleum dependent polymer, was reinforced with carbon black and other non-petroleum dependent fillers. Blends of carbon black with these fillers were found to be satisfactory for tank track pad applications.		

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ACKNOWLEDGEMENTS

We wish to express thanks to B.F. Goodrich and Polysar Companies for making available their Laboratory facilities to perform some of our tests.

The authors are also indebted to the Standard Products Laboratory personnel for their devoted effort to successfully complete this project.

Special thanks are expressed to the U. S. Army for providing financial support for this project.

SCOPE

The objective of this work was to develop rubber tank track pads based on Guayule rubber and lignin or other non-oil dependent fillers yielding equal or better service than current pads.

INTRODUCTION

Current military rubber tank track pads are predominantly composed of petroleum derived materials. Possible emergency shortages of petroleum indicate a need for the study of potential substitutes.

Guayule rubber from domestic sources is a potential replacement for the petroleum derived SBR rubber, which comprises more than 50% by weight of current tank track pads. It is also a potential substitute for the natural rubber (1 - 7), which has been shown to give excellent abrasion and tear resistance in tank track pads (8).

Reinforcing carbon black, also a petroleum derivative constituting approximately 30% by weight of the tank track rubber, is the second largest fraction and an important part of the tank track pad. Potential substitutes for carbon black include lignin (wood by-product) (9 - 12), silica derivatives including Fillite and Orbaloid, silane-modified kaolin (Nucap), a coal by-product (Austin Black), and chopped cellulose fibers; such as, Santoweb D and DX (13). It is the intention of this work to compound such non-oil components of rubber and filler and make tank track pad performance equal to or better than those currently in service.

APPROACH

Experimental:

Rubber compounds were mixed in a laboratory size internal mixer (Farrel B) and sheeted out on a 12-inch rubber mill. The latter was also used to remill the compounds 24 hours after mixing. Tensile strength, elongation, and modulus were determined by employing an Instron Model 1125 runner tensile tester equipped with an Instron Model A74-80 chamber for low and elevated temperature testing.

Abrasion resistance was determined using a Pico Abrader (8). Flex crack growth resistance was evaluated on a DeMattia Flex Tester (1). Hysteresis data were obtained with a Goodrich Flexometer (2). Chunking and chipping resistance was evaluated with a tester developed by Polysar, Ltd. of Canada (3,14). Cure rate data were determined with a Monsanto Model 100 oscillating disc rheometer. Scorch (processing safety) and plasticity data were developed with a Monsanto Mooney Tester.

All physical properties were determined by ASTM procedures where applicable. Physical properties of experimental compounds were compared with proprietary commercial SBR compounds which are currently in use on military equipment.

In addition to the cure rate, scorch, and plasticity data, processing characteristics such as power consumption, heat development, and polymer breakdown were determined by using a Haake Laboratory internal mixer (Rheocord Model 600). Abbreviations of testing conditions for various Laboratory instruments are given in the Appendix.

Properties of Materials:

The Guayule rubber for this work was obtained from the Mexican Government operated pilot plant located in Saltillo, Coahuila in northern Mexico. This rubber was found to have a Specific Gravity of 0.92, Volatile Matter 0.31%, Acetone Extract 5.86%, Ash Content 0.455% and Mooney Viscostity (ML4/212 Deg. F.) - 79.

The rubber appeared to be stickier than the SIR-20 (16) natural rubber also used in this study and contained trace amounts of fiber which were not apparent after mixing and did not show up in any of the tensile, tear, or flex fractures. In Figure 1 the molecular weight distribution of the polymer is shown for the two sets of polymers received. Figure 2 shows the Infra-red Spectrum obtained for the type of guayule rubber used. In Table 1 a comparison of the properties of the guayule rubber and the natural rubber is also shown, as measured in the Rheocord internal mixer. The results shown in Figure 3 indicate that the processing of the guayule rubber is better than or equal to the natural rubber.

The Indulin AT lignin used was obtained from the Chemical Division of Westvaco. Indulin AT is made as a by-product of the Kraft process for paper pulp. The Westvaco data bulletin (Table 2) gives the properties for this product. Indulin AT contains about 5% moisture. Therefore, it was necessary to dry the lignin in a circulating air oven for a minimum time of four hours at 158° F. before mixing the compounds containing lignin.

Fillite is the tradename of Fillite, Ltd., Runcorn, U.K. The material evaluated had a specific gravity of 0.7 and consists of hollow silicate microspheres. In Table 3 the material characteristics for the Fillite are depicted.

Orbaloid TM is complex aluminum silicates in the form of pyrogenic microspheres with a specific gravity of 2.04. In Table 4 the physical characteristics of the material used are shown.

Nucap 200 TM from J.M. Huber Company is silane-treated, hydrated aluminum silicate. This material has a specific gravity of 2.6 and a mean particle size of 0.3 microns.

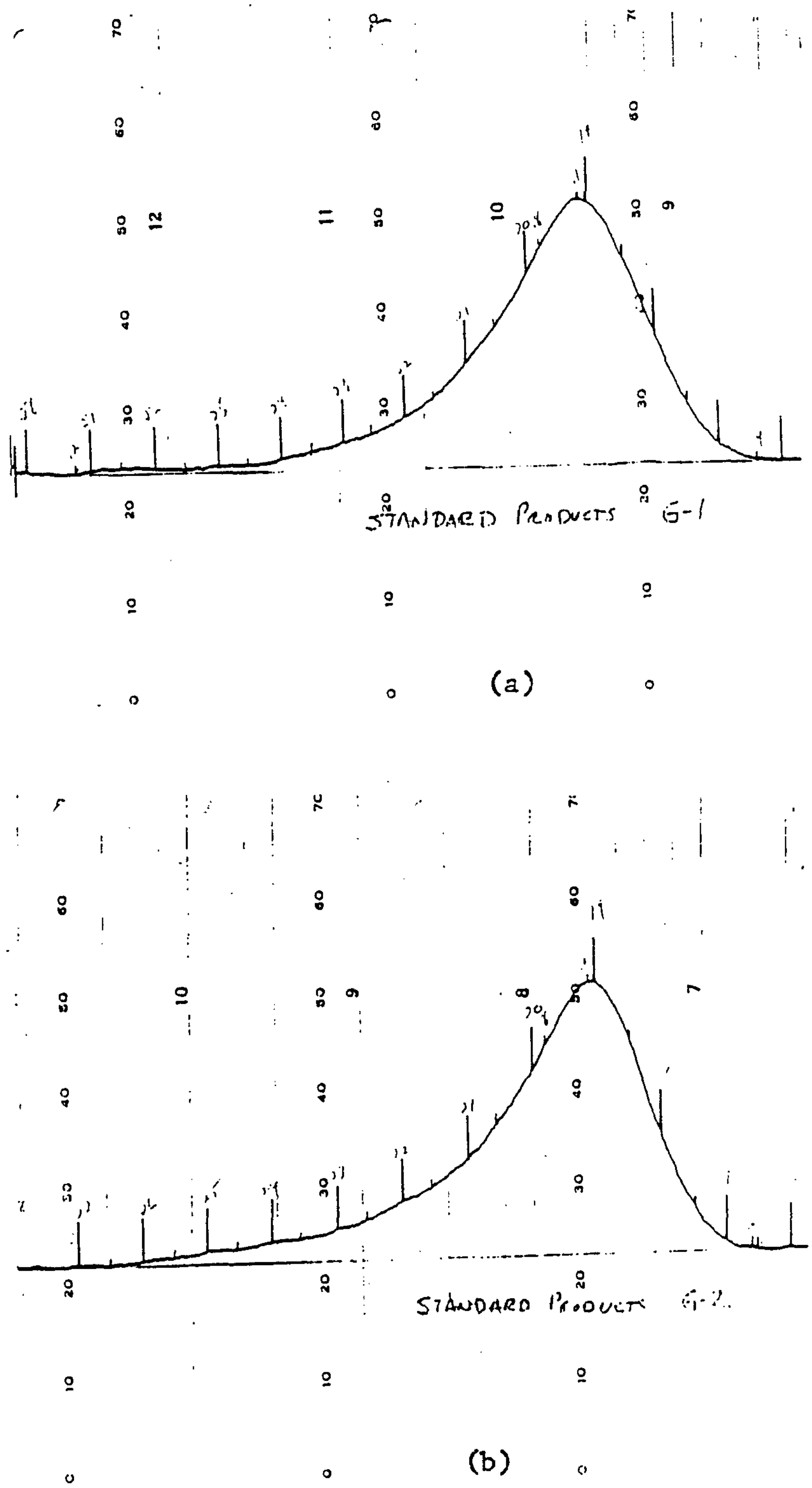
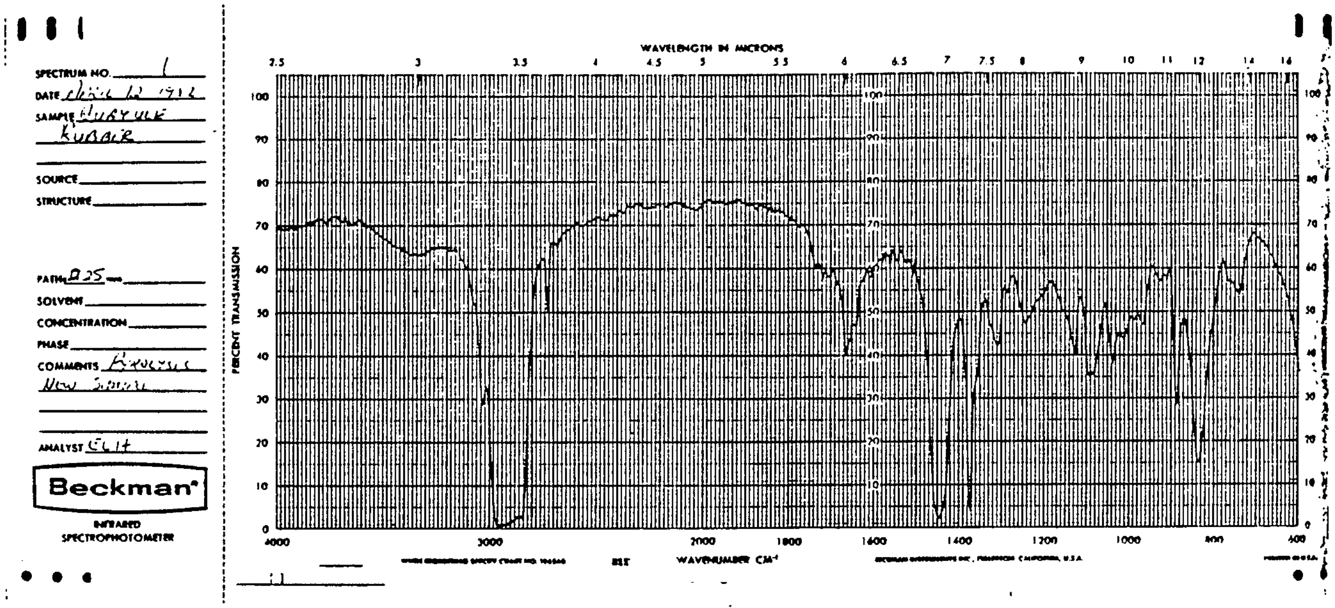


Figure 1. GPC curves for the Guayule Rubber (a) Sample 1; Mn = 526.7 X 10 $^{-3}$; Mn = 1626.7 X 10 $^{-3}$; (b) Sample 2; Mn = 511.4 X 10 $^{-3}$; Mw = 1407.1 X 10 $^{-3}$



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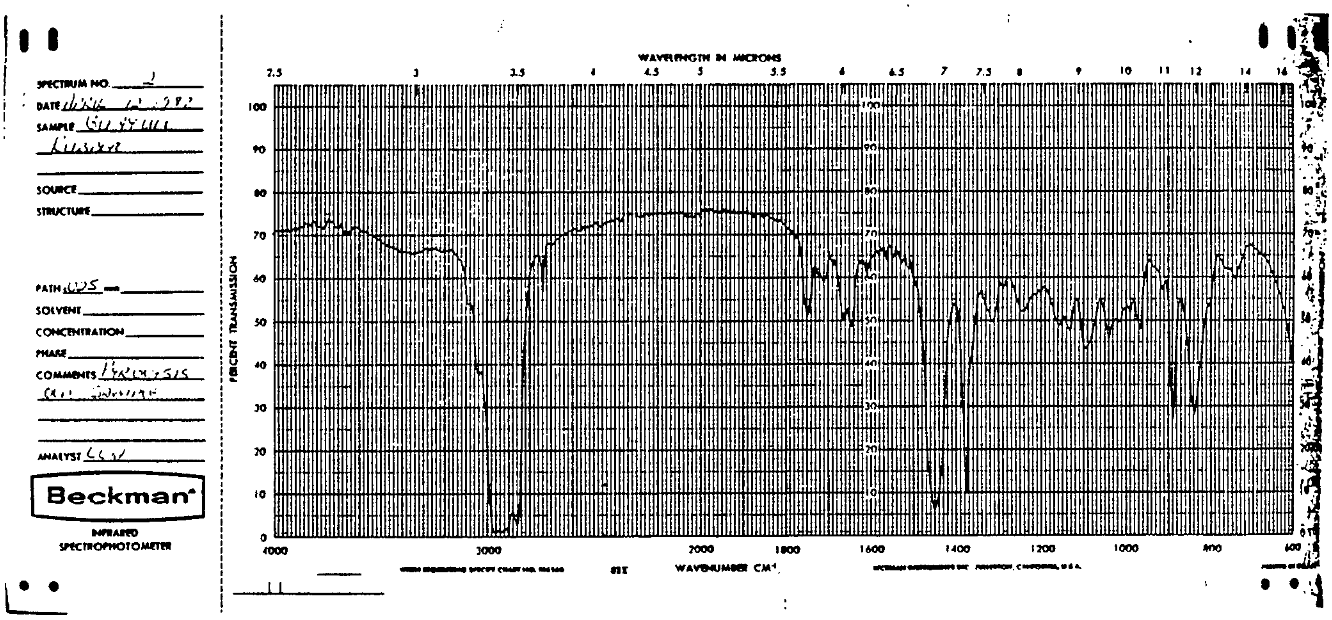


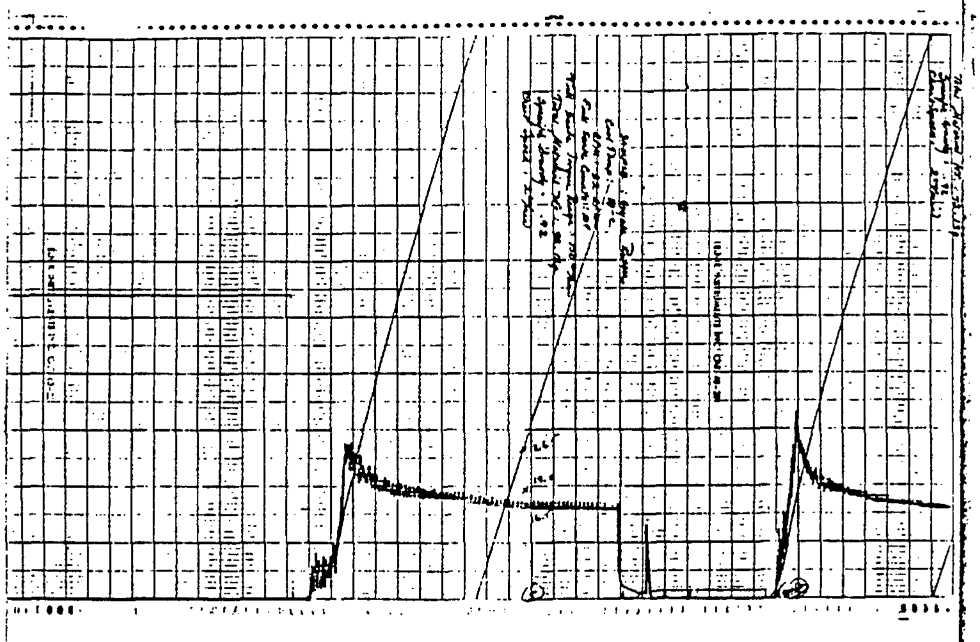
(b)

Figure 2. Infra Red Spectra for Guayule Rubber (a) Sample 1 (b) Sample 2

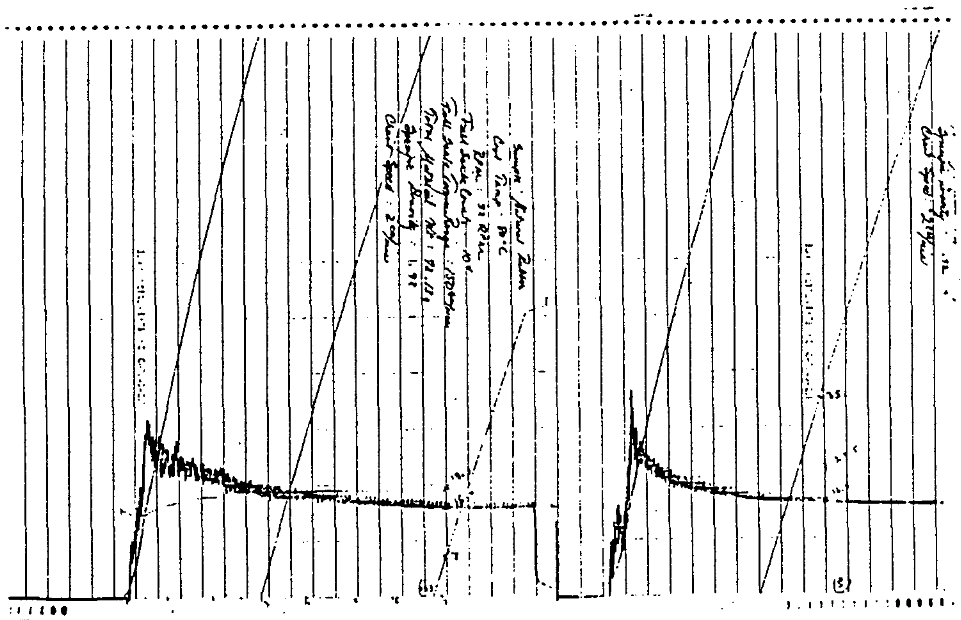
TABLE 1: RHEOLOGICAL PROPERTIES OF THE GUAYULE AND SIR-20 NATURAL RUBBER

	RPM	TIME (MIN)	ENERGY (MJ/m^3)	TORQUE (Nm)	Temp. C
Guayule Rubber	32	5.0	325.6	24.8	97.5
	64	4.8	630.8	24.0	110.5
	128	5.5	1364.1	24.8	150.0
Natural Rubber	32	7.0	530.8	24.0	96.5
	64	5.0	692.3	24.8	112.5
	128	6.0	1461.5	23.2	150.0

* Above Data Were Obtained From Rheographs Typically Shown In Figure 3 When Temperature Was In Equilibrium.



(a)



(b)

Figure 3. Representative Rheocord Graphs for (a) Guayule and (b) Natural Rubber

TABLE 2 PHYSICAL PROPERTIES OF LIGNIN

INDULIN AT

PRODUCT DESCRIPTION:
Kraft Pine Lignin Polymer

LIGNOCHEMICAL DATA BULLETIN

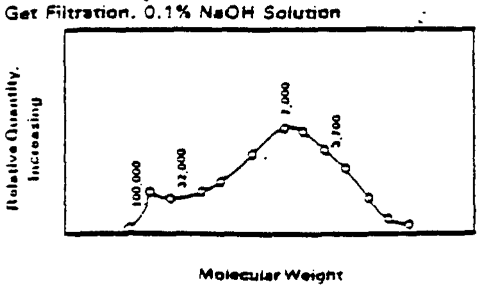
Typical Properties

Physical Form	Free Flowing Brown Powder
Lignin content, % (on dry basis)	99
pH of 2% aqueous slurry at 25°C	6
Insolubles in warm 5% NaOH solution, %	0.05
Surface tension of 1% solution of sodium salt of INDULIN AT, dynes/cm	43
Flash point, °F	349
Fire point, °F	389
Sintering temperature, °F	370
Weight loss on heating in air, %	
300°F	1
400°F	7
500°F	40
600°F	75
Bulk density, lbs./ft.^{3}, loose	26
packed	32
Ash, % (on dry basis)	1
Moisture, %	5
Specific gravity	1.3

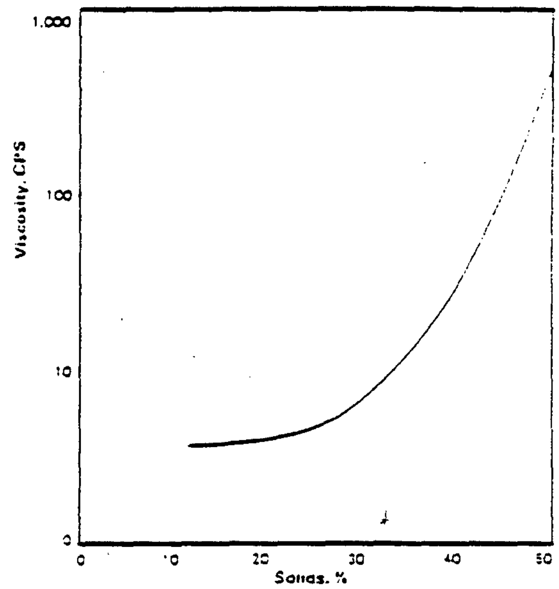
Transportation and Use Data:

DOT Shipping Class .... Non Hazardous
LD_{50} .... >12g/kg
Minor eye irritant, not irritating to skin
TSCA:
CAS number 8068-05-1
EPA number R244-7025
Exempt from the requirement of a tolerance in or on raw agricultural commodities (40 CFR, sec. 180.1001).

Molecular Weight—Distribution—Gel Filtration, 0.1% NaOH Solution



Viscosity of Aqueous Slurry, at 25°C, pH 12



Solubility in organic solvents

Benzene—nil
Hexane—nil
Methyl Ethyl Ketone—Partial
Methyl alcohol—Partial
Ethylene glycol—complete
Dioxane—complete
Monoethanolamine—complete
Dimethyl formamide—complete
Cellosolve—complete
50:50 Ketone Alcohol—complete

*PACKAGING—multiwall paper bags containing 50 lbs. net weight.

Westvaco
Chemical Division
Polychemicals Department
P.O. Box 5207
North Charleston, S.C. 29406
U.S.A.
Telephone: 803-554-8350
TWX: 810-881-1876

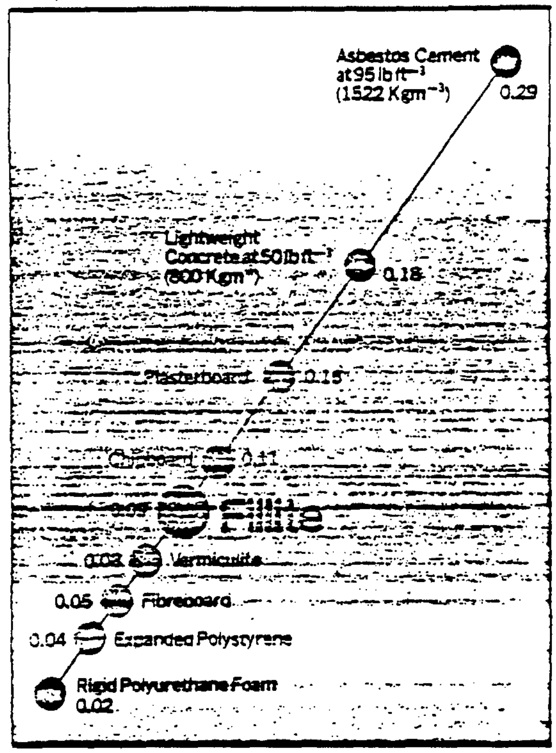
TABLE 3 MATERIAL CHARACTERISTICS OF FILLITE

Typical Applications	Sphere Density	Processes
Friction (Non slip surfaces) Abrasion resistance.	0.82	High Shear mixing operations
Lightweight concretes. Sealing mastics.	0.7	General plastics processing routes. Injection moulding.
Adhesives Putties.	0.6	Compression moulding, pultrusion, filament winding, resin injection, spray-up, cold press moulding, rotational casting, hand lay-up.
Adhesives	0.5	
Sandwich cores of low dielectric loss	0.4	Speciality materials generally involve in low shear fabrication processes such as casting, trowelling etc.
Buoyancy	0.35	
Radomes		
Additionally available as Metal Coated with Au, Ag, Pt, Cu.	0.3	Surface treated grades of all densities are available with a variety of coupling agents.

For accurate cost comparisons of Fillite with other fillers the appropriate density ratio should be used. For example, with a filler at a density of 2.8 and Fillite at a density of 0.7 the ratio is 4.

As a general rule, the average sphere diameter falls as the density is reduced, from 200 micron for 0.82 to 50 micron for 0.4 and below.

Comparison of Materials 'K' Factors $Wm^{-1}K^{-1}$



The 'K' value of a material is an extremely important measure of its thermal insulating capacity. The lower this number the better is the insulation of the material. Values for various materials are shown in the graph above.

Material Characteristics

Description

Form	:	Free Flowing Hollow Microsphere
Colour	:	Off White
Surface Moisture	:	0.5% maximum

Physical Properties

Density Range	:	0.3—0.82
Bulk Density	:	0.18—0.45
Packing Factor	:	60%/62%
Hardness	:	Moh's Scale 5
Average Wall Thickness	:	1/10 sphere diameter
Failure (Hydrostatic)	:	Implosion

Electrical Properties

With Fillite 0.7 the relative permittivity may be taken as 2.2 and the loss tangent 0.024 (both measurements at 50 Hz).

Chemical Properties

Composition:

Shell Material:	Alumina	(As:	Al_2O_3) 26%—30%
	Silica	(As:	SiO_2) 25%—35%
	Iron	(As:	Fe_2O_3) 4% maximum
Gas:	Nitrogen		30%
	Carbon Dioxide		70%
	Total pressure		0.2 std. atm.

Thermal Properties

Melting Temperature: 1200°C—1350°C
Thermal Conductivity: 0.09 $Wm^{-1}K^{-1}$
Co-efficient Thermal Expansion: $\propto = 8 \times 10^{-6}$ (°K)

Health & Safety

Fillite is classified as a Nuisance Dust similar to talc, rouge, etc. Long term effects of continued exposure is minimised as the spheres below 5 microns have been removed.

TABLE 4 MATERIAL CHARACTERISTICS OF ORBALOID

ORBALOID

A NEW MINERAL FILLER

PRODUCT DESCRIPTION:

Greyish colored pyrogenic microspheres consisting principally of complex aluminum silicates. This spherical filler is a superior extender for rubber, plastics, coatings, sealants, and adhesives.

PARTICLE DESCRIPTION:

Essentially spherical particles with an average size of 30 microns and a range of 5 microns to 45 microns: mean particle size - 15 microns.

SIEVE ANALYSIS:

100% (±1%) thru 200 mesh screen
95% (±1%) thru 325 mesh screen
ASTM # (C-430)

SPECIFIC GRAVITY:

17.0 LBS / gallon
2.04 specific gravity

AVERAGE BULK DENSITY:

8.4 LBS / gallon
48-50 LB / cubic foot
.0588 Bulking Valve

OIL ABSORPTION FACTOR:

Equated as the # of lbs of oil per 100 lbs.
4.5 oil absorption

ANALYSIS:

Silica as S_i0_2	45% - 55%
Aluminum as $A1_20_3$	27% - 36%
Iron as Fe_20_3	5% - 8%
Calcium as C_a0	0.1% - 0.8%
Magnesium as Mg0	0.2% - 1.0%
Free Carbon as C	◄6%
PH Factor	6.5% - 7.0%

HANDLING PROCEDURE:

Provide adequate ventilation to minimize release of dust to the environment: Equipment available thru Midwest Filler Products.

Austin Black is the tradename of the Slab Fork Coal Company. The material is comprised of bituminous fine black with a specific gravity of 1.22 and an average particle size of 0.3 microns.

Santoweb TM (Monsanto) consists of short, surface treated cellulose fibers designed for the reinforcement of rubber vulcanizates.

HiSil TM is hydrated silica from PPG which is used for the reinforcement of polymers. HiSil 210 was used in this study. The material has a specific gravity of 2.0 and an ultimate particle size of 0.022 microns.

<u>Selection of Cure System:</u>

Guayule rubber was difficult to obtain in large quantities. It was, therefore, necessary to use natural rubber in the screening of the various cure systems. Most of the preliminary studies were made by utilizing SIR 20 natural rubber, which was found to yield comparable to slightly improved vulcanizates.

Several variations of commercial compounds were compared to select an optimum formula for the evaluation of carbon black replacements in guayule. These formulae are listed in Table 5 and comparative properties are listed in Tables 6 to 9. Formula D produced the best balance of properties for both guayule and natural rubber. The lower hardness and modulus produced by carbon black replacements studied indicated that elimination of the process oil from Formula D would be beneficial. The resulting Formula F tends to optimize the vulcanizate properties of the guayule and the less reinforcing substitutes (for carbon black) without seriously affecting the vulcanizate properties of natural rubber and carbon black, which appear to involve less critical optima.

All systems of rubber/filler used in this study were cured with the F system as discussed in the next sections.

TABLE 5: BASE FORMULAE USED FOR THE SELECTION OF THE CURE SYSTEM

No. (Prefix)	A	B	C	D	E	F	G	H
Rubber	100	100	100	100	100	100	100	100
Total Reinforcement	62	62	62	62	62	62	62	62
Zinc Oxide	4	15	5	3.5	3	3.5	3	3
Stearic Acid	1.5	2	2	3	2	3	3	2
Sundex 790	10	3	8	13	6	---	---	---
Piccopale 100	---	---	---	---	3.5	---	---	---
Sunolite 240	1	---	1	---	1	---	---	---
Sunolite 666	3	---	---	3	---	3	3	3
Flectol H	1	1	1	1	2.5	1	1	1
UOP 688	---	---	---	---	5	---	---	---
Santoflex 13	2	2	2	2	---	2	2	2
Sulfur	1.6	3	2.5	1.25	2	1.25	1.25	1.25
TBBS	1.1	0.6	1.0	1	1.45	1	---	---
OBTS	---	---	---	---	---	---	1.25	1.50
	187.90	188.60	182.60	189.75	186.45	176.75	176.50	176.75

TABLE 6: PRELIMINARY PROPERTIES OF SBR-, NATURAL RUBBER-, AND GUAYULE-FILLER COMPOUNDS USING CURE SYSTEM A

COMPOUND NO.		A-1	A-2	A-3	A-4	A-5	A-6	A-7	A-8
POLYMER		SBR	SBR	SBR	SBR	NR	NR	GR	GR
CARBON BLACK/FILLER		N234	N330	LIGNIN	FILLITE	N234	N330	N234	N330
ML4/212°F		69	53	36	36	80	67	79	62
MS 250°F	T4	55	49	60	37	18	22	22	25
	Min	28	21	11	11	38	30	36	23
SPECIFIC GRAVITY		1.14	1.15	1.04	1.03	1.13	1.14	1.14	1.14
SHORE A		73	71	51	53	69	68	68	66
MOD - 100		500	540	125	100	425	490	440	390
MOD - 200		1400	1500	160	120	1300	1210	1200	1050
MOD - 300		2300	2425	200	140	2300	2275	2050	1750
TENSILE (PSI)		3130	2800	530	190	3750	3350	3650	3400
ELONGATION		350	345	700	415	465	430	500	525
REBOUND		25	27	49	47	28	33	23	27
TEAR DIE C		221	242	61	40	643	647	595	620
COMP. SET METHOD B	(-30)	55	47	37	26	46	38	53	48
	(72)	20	17	14	12	20	16	21	16
	(212)	41	38	41	62	41	38	39	37

CURED 25 MINUTES @ 298°F

TABLE 7: PRELIMINARY PROPERTIES OF NATURAL RUBBER-, AND GUAYULE-FILLER COMPOUNDS

Compound No.	B-1	B-2	B-3	B-4	C-1	C-2	C-3	C-4	C-5	C-6
Polymer	NR	NR	GR	GR	NR	NR	NR	GR	GR	GR
Carbon black	N234	N330	N234	N330	N110	N234	N330	N110	N234	N330
ML4/212°F	96	78	90	54	84	97	79	96	93	55
MS 250°F T4	32	30	35	46	20	16	23	30	22	29
Mn	43	34	42	30	36	41	35	36	36	27
Specific Gravity	1.21	1.21	1.21	1.21	1.14	1.13	1.14	1.14	1.14	1.14
SHORE A	72	72	71	71	76	76	74	76	76	73
Mod 100	825	925	600	675	775	925	775	675	850	775
Mod 200	2125	2200	1650	1675	1900	2250	1850	1650	1925	1850
Mod 300	3250	3175	2650	2550	3100	3425	2800	2624	2900	2800
Tensile (PSI)	3675	3225	3650	3275	3800	3750	3300	3650	3725	3350
Elongation	350	335	415	400	380	340	335	440	380	375
Rebound %	31	35	26	29	34	35	37	29	31	32
Tear Die B	618	425	655	603	568	661	650	679	650	544
C. Set (-20F)	24	18	44	36	32	32	30	28	29	33
(72F)	13	9	15	14	13	12	8	16	14	12
(212F)	64	54	61	58	54	51	44	59	55	55

Cured 25 Minutes at 298°F

TABLE 8: PRELIMINARY PROPERTIES OF POLYBUTADIENE - (BR), SBR-, NATURAL RUBBER-, AND GUAYULE-FILLER COMPOUNDS USING CURE SYSTEM D

COMPOUND NO.		D-1	D-2	D-3	D-4	D-5	D-6	D-7	D-8	D-9	D-10	D-11	D-12
GUAYULE		--	--	--	--	--	--	60	60	60	60	100	100
NATURAL		60	60	60	60	100	100	--	--	--	--	--	--
BR		--	--	40	40	--	--	--	--	40	40	--	--
SBR		40	40	--	--	--	--	40	40	--	--	--	--
CARBON BLACK		N234	N330	N234	N234	N234	N330	N234	N330	N234	N330	N234	N330
ML4/212 °F		51	73	72	51	76	61	65	51	76	67	69	60
MS 250°F	T4	47	38	36	43	25	27	50	50	58	60	41	45
	Mn	20	37	36	22	35	27	29	23	31	24	29	26
SHORE A		65	69	67	69	69	65	67	65	66	54	60	64
MOD - 100		375	325	350	350	375	400	250	250	250	250	250	225
MOD - 200		925	900	950	925	1025	1100	600	550	650	600	650	600
MOD - 300		1700	1750	1775	1700	1900	2025	1175	1100	1350	1175	1300	1200
TENSILE (PSI)		3000	3300	3300	3025	3450	3575	3050	3000	3225	2900	3275	3175
ELONGATION		500	510	495	490	495	485	615	620	565	580	590	615
TEAR DIE C		433	645	506	514	693	713	590	480	560	615	713	600
C. SET	(-30 F)	75	81.5	70.0	74.5	68.5	59.5	84.4	79.4	76.2	72.9	72.7	71.9
METHOD B	(72 F)	13.6	16.9	14.0	10.7	16.4	9.8	24.5	22.0	13.0	15.5	20.6	15.5
	(212 F)	41.3	47.2	49.3	49.8	49.8	40.4	46.5	46.5	49.0	46.0	47.0	42.5

Cured 25 Minutes at 298°F

TABLE 9: PRELIMINARY PROPERTIES OF POLYBUTADIENE-(BR), SBR-, NATURAL RUBBER-, AND GUAYULE-FILLER COMPOUNDS USING CURE SYSTEM E

COMPOUND NO.		E-1	E-2	E-3	E-4	E-5	E-6	E-7	E-8	E-9	E-10
GUAYULE		--	--	--	--	--	60	60	60	100	100
NATURAL		60	60	60	100	100	--	--	--	--	--
BR		28	28	28	--	--	28	28	28	--	--
SBR		12	12	12	--	--	12	12	12	--	--
CARBON BLACK		N110	N234	N330	N234	N330	N110	N234	N330	N234	N330
ML4/212°F		72	75	57	77	62	67	74	56	75	64
MS 250°F	T4	21	23	28	18	24	30	29	33	23	28
	Mn	33	34	25	27	29	30	33	24	33	28
MOD - 100		425	475	500	400	475	350	400	350	375	350
MOD - 200		1100	1275	1275	1250	1225	850	1000	900	1075	925
MOD - 300		1975	2250	2125	2175	2050	1680	1800	1600	1925	1600
TENSILE (PSI)		3350	3450	3125	3675	3375	3375	3375	2875	3625	3425
ELONGATION		475	450	440	475	480	550	515	500	540	565
TEAR DIE C		520	664	270	545	537	420	455	344	617	510
C. SET METHOD B	(-30°F)	66.5	64	55	68.5	43.5	79.1	76.8	65.5	62.9	51.8
	(72°F)	13.0	14.4	9.8	12.0	8.6	13.5	13.0	9.0	13.0	10.0
	(212°F)	42.0	43.1	39.3	44.7	43.4	55.0	57.0	59.0	59.0	56.0

Cured 25 Minutes at 298°F

RESULTS AND DISCUSSION

Several comparisons of the fillers have been used with natural rubber, guayule rubber and blends of the same to determine the optimum of the rubber/filler blend system to yield physical properties comparable to a compound used currently in track pads. The results of such combinations are tabulated and discussed separately on a filler by filler basis.

A. Guayule and Natural Rubber Reinforced With Lignin

Lignin was evaluated as a complete and a partial replacement for carbon black in the guayule and the natural rubber tank track pad control compounds containing N234 carbon black. These compounds were evaluated to determine the effect on the processing and physical properties of these compounds. The results were also compared to a current SBR tank track pad compound (SP-1).

In the guayule compounds, total replacement of the carbon black with lignin produced significantly improved processing characteristics as indicated by lower plasticity and longer scorch times compared to both the guayule carbon black control and the current SBR track pad compound shown in Table 10. Partial replacement of the carbon black by lignin resulted in equal to or slightly improved processing characteristics in both the natural and the guayule rubber compounds.

Total replacement of the carbon black by lignin in the guayule compound decreased the hardness and the modulus and produced a severe adverse effect on tensiles. Partial replacement by lignin (25%) produced lesser effects in the same direction. In the latter compound, added lignin partially restored the hardness and modulus, but produced negative effects on the tensile strength and tear resistance. Moderate to severe adverse effects were also noted for partial to complete replacement of

TABLE 10: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH LIGNIN.

COMPOUND		CURE*	SP-1	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
Guayule			(SBR)	--	100	100	100	100	--	--	--
Natural			--	100	--	--	--	--	100	100	100
N234 Black			--	62	62	--	47	47	47	47	--
N110 Black			--	--	--	--	--	--	--	--	47
Lignin			--	--	--	43	11	22	11	22	11
ODR 370°F											
Min			11.1	27.5	18	8	18	21	17.5	19.5	16.0
Max			97.9	89.3	65	46	62	59	71.0	69.0	71.0
T4			1.6	.9	.9	1.9	1.1	.9	1.0	1.0	1.1
Tc			2.8	1.9	1.8	3.0	2.0	1.9	2.0	2.0	2.1
ML1+4/212°F			50		67	30	53	59			
MS at 250°F											
T4			48	10.8	12	35	14	13	15.5	15.5	22.0
Min			21.8	50.0	34	13	29	34	32.2	34.5	28.0
MODULUS - 100 (PSI)	-30°F	30	1400	720	625	210	440	480	395	--	--
	72°F	10	640	485	450	90	300	350	365	365	315
		20	660	520	475	150	390	440	400	385	385
		30	600	570	495	140	375	420	365	415	370
	250°F	30	440	335	295	130	230	240	205	--	--
MODULUS - 200 (PSI)	-30°F	30	--	1580	1415	235	965	965	1045	--	--
	72°F	10	1650	1410	1325	170	710	770	790	800	785
		20	1725	1510	1390	310	940	930	950	865	900
		30	1600	1550	1365	260	875	915	900	890	850
	250°F	30	995	660	575	170	440	390	350	--	--

TABLE 10. CONT.

COMPOUND		SP-1	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
MODULUS - 300 (PSI) -30°F	30	--	2250	2125	820	1665	1600	1785	--	--
72°F	10	2650	2420	2300	285	1300	1340	1635	1465	1500
	20	2775	2630	2400	460	1615	1590	1750	1585	1635
	30	2620	2660	2350	505	1640	1530	1650	1660	1565
250°F	30	--	1180	1015	195	540	560	550	--	--
MODULUS - 400 (PSI) -30°F	30	--	--	--	1470	--	--	--	--	--
72°F	10	3300	3250	3025	490	1975	2000	2490	2250	2350
	20	--	3500	3360	780	2390	2350	2645	2500	2500
	30	--	3550	3180	800	2425	2265	2510	2465	2400
250°F	30	--	1710	1505	220	840	770	830	--	--
TENSILE (PSI) -30°F	30	2625	2670	2600	1980	2300	2150	2730	--	--
72°F	10	3300	3938	3775	1200	3400	2820	3540	2865	3365
	20	3110	4017	3665	2100	3600	3025	3500	3000	3000
	30	3100	4038	3835	2200	3475	3025	3500	2985	2900
250°F	30	1350	2410	2300	780	1750	1500	2070	--	--
ELONGATION -30°F	30	170	370	360	455	385	380	390	485	525
72°F	10	400	490	520	560	600	540	510	465	510
	20	340	467	480	590	560	500	500	485	510
	30	370	465	500	600	530	500	510	--	--
250°F	30	265	515	560	900	660	715	790	--	--
SHORE A 72°F	10	75	68	65	39	64	65	65	65	64
	20	76	69	70	46	65	67	66	66	66
	30	76	69	70	48	65	68	66	66	66
TEAR DIE B (Lb/In) -30°F	30	--	--	--	--	--	--	--	--	--
72°F	10	359	921	745	120	758	494	792	571	827
	20	299	729	781	191	740	555	734	550	716
	30	356	669	777	107	742	604	625	539	686
250°F	30	135	596	417	128	360	325	341	--	--

TABLE 10: CONT'D.

COMPOUND	SP-1	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
AGED 70 HRS./158°F									
MODULUS - 100	750	700	650	175	510	575	510	--	--
MODULUS - 200	1900	1880	1775	340	1085	1115	1150	--	--
MODULUS - 300	2900	3000	3075	575	2090	2000	2020	--	--
MODULUS - 400	--	3850	3725	1000	2515	2625	2925	--	--
TENSILE (PSI)	3140	3850	3875	2360	3215	3040	3550	--	--
ELONGATION	335	400	420	575	485	460	480	--	--
DEMATTIA FLEX (kc/0.1 in)	3	41	47	4	14	8	21	--	--
BFG FLEXOMETER									
TEMP (3 MIN)	61	68	86	39	68	104	88	--	--
TEMP (6 MIN)	80	89	108	71	86	80	110	--	--
MIN TO BLOW	30+	7	6	7	15	6	6	--	--
PICO ABRADER	122	177	153	22	85	66	97	--	--
COMPRESSION SET, %									
-30°F	79	76	72	69	82	88	70	--	--
72°F	15	10	11	5	10	11	10	--	--
212°F	27	47	46	45	45	44	48	--	--
CHIPPING & CHUNKING	0	2	4		9				

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)
(SP-1 Cured At 320°F)

carbon black (by lignin) in the elevated temperature properties of modulus, tensile strength, and tear resistance. Similar effects were produced by lignin in the natural rubber compounds.

The all-lignin reinforced compound appeared to produce poor flex cut growth and a low hardness. However, the partial replacement of carbon black by lignin improved this property. The all-lignin reinforced compound did not improve the time to blowout despite the fact that this compound evidenced much lower heat generation. Abrasion resistance was severely effected in proportion to the level of lignin in the compounds. Lignin was also evaluated at the 25 percent level utilizing a more reinforcing carbon black (N110). However, the properties of this compound evidenced little improvement over the results obtained with 25 percent lignin and N234 carbon black.

B. Guayule and Natural Rubber Reinforced with Fillite

Fillite produced a drastic reduction in modulus and tensile strength compared to carbon black (N234) in an SBR tank track pad compound, as shown in Table 6. Therefore, it was necessary to evaluate this filler at different levels in the guayule and natural rubber compounds to study its reinforcing effects. Table 11 summarizes the results of the physical properties obtained for the different compounds mixed.

Twenty-five percent replacement of the N234 carbon black with Fillite in the guayule tank track pad control compound produced a desirable effect on the processing properties. A moderate reduction of hardness, modulus, and tensile strength was also measured at ambient temperature. Little effect was noticed on ambient tear strength. At elevated test temperatures, the modulus, tensile and tear strength were slightly reduced. Flex cut growth (Demattia) was also reduced.

TABLE 11: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH FILLITE

COMPOUND		CURE*	F-1	F-9	F-10	F-11	F-12
Guayule			--	--	--	--	100
Natural			100	100	100	100	--
N234 Black			62	47	47	--	47
N110 Black			--	--	--	47	--
Fillite			--	6	12	6	6
ODR 370°F	Min		27.5	21.2	18.0	19.5	16.0
	Max		89.3	80.5	81.3	79.0	63.0
	T4		.9	1.1	1.0	1.0	1.0
	Tc		1.9	2.1	2.0	2.0	1.9
ML1+4/212°F			--	--	--	--	57
MS at 250°F	T4		10.8	14.6	16.6	16.8	18
	Min		50.0	33.7	30.7	31.6	25
MODULUS - 100 (PSI)	-30°F	30	720	375	--	--	375
	72°F	10	485	315	340	325	310
		20	520	360	360	350	345
		30	570	365	360	340	360
	250°F	30	335	230	--	--	335
MODULUS - 200 (PSI)	-30°F	30	1580	925	--	--	960
	72°F	10	1410	800	840	800	810
		20	1510	980	940	900	890
		30	1550	1025	940	825	930
	250°F	30	660	440	--	--	445
MODULUS - 300 (PSI)	-30°F	30	2250	1680	--	--	1750
	72°F	10	2420	1740	1600	1520	1515
		20	2630	1975	1800	1700	1650
		30	2660	1975	1800	1625	1730
	250°F	30	1180	735	--	--	730
MODULUS - 400 (PSI)	-30°F	30	--	2500	--	--	2600
	72°F	10	3250	2540	2415	2375	2320
		20	3500	2940	2700	2650	2510
		30	3550	3020	2700	2560	2530
	250°F	30	1710	1100	--	--	1065
TENSILE (PSI)	-30°F	30	2670	2500	--	--	2600
	72°F	10	3938	3750	3300	3700	3565
		20	4017	3625	3250	3550	3575
		30	4038	3565	3200	3700	3550
	250°F	30	2410	1270	--	--	1860

TABLE 11: CONT.

COMPOUND		F-1	F-9	F-10	F-11	F-12
ELONGATION	CURE*					
-30°F	30	370	400	--	--	400
72°F	10	490	545	500	560	560
	20	467	560	485	500	530
	30	465	560	460	530	510
250°F	30	515	450	--	--	600
SHORE A						
72°F	10	68	58	60	61	63
	20	69	61	61	62	64
	30	69	61	61	62	64
TEAR DIE B (LB/IN)						
-30°F	30	--	--	--	--	--
72°F	10	921	771	648	831	858
	20	729	700	703	806	835
	30	669	768	656	751	745
250°F	30	596	326	--	--	307
AGED 70 HRS/158 F						
MODULUS - 100		700	450	--	--	400
MODULUS - 200		1880	1150	--	--	1140
MODULUS - 300		3000	2130	--	--	2020
MODULUS - 400		3850	3170	--	--	2940
TENSILE-(PSI)		3850	3780	--	--	3600
ELONGATION		400	470	--	--	490
DEMATTIA FLEX (kc/0.1 in)		41	25	--	--	22
BFG FLEXOMETER						
TEMP (3 MIN)		68	56	--	--	72
TEMP (6 MIN)		89	64	--	--	83
MIN TO BLOW		7	27	--	--	15
PICO ABRADER %		177	99	--	--	69
COMPRESSION SET, %						
-30°F		76	67	--	--	78
72°F		10	9	--	--	10
212°F		47	49	--	--	41

* CURE TIME IN MINUTES AT 298°F

(30 Minutes where not indicated)

The partial Fillite replacement of the carbon black appeared to reduce the heat generation and significantly increased the time to blowout. However, abrasion resistance was considerably reduced. Fillite produced comparable results in the natural rubber control tank track pad compound.

The two compounds evaluated in an effort to increase the modulus to the level of the natural rubber control compound produced the following results. The compound with added Fillite failed to restore the 300% modulus, but did not affect significantly the tensile and tear strength. A second compound containing a more reinforcing carbon black (N110) and 25% Fillite also failed to increase the modulus, but appeared to have a slight beneficial effect on ambient tear strength.

C. Guayule and Natural Rubber Reinforced with Orbaloid

Table 12 shows that processing characteristics have improved with the replacement of carbon black by Orbaloid in both Guayule and Natural Rubber compounds. This is indicated by longer scorch times and lower Mooney viscosities.

Hardness, modulus, tensile strength and tear resistance were slightly decreased at low, ambient and elevated temperatures by the partial replacement of carbon black with Orbaloid in a Guayule tank track pad vulcanizate. Flex cut growth and abrasion resistance were significantly reduced, but the time to blowout and the heat generation characteristics were significantly improved.

Increase of Orbaloid in the compound with guayule rubber increased the hardness, but further reduced the tear resistance and tensile strength at all test temperatures. Abrasion resistance was further reduced along with the advantage in blowout time and heat generation.

The above results parallel the observations from studies with natural

TABLE 12: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH ORBALOID

COMPOUND		CURE*	F-12	F-13	F-14	F-15	F-16	F-17	F-18	F-19
Guayule			100	--	--	--	--	--	100	100
Natural			--	100	100	100	100	100	--	--
N234 Black			62	47	31	16	--	47	47	47
N110 Black			--	--	--	--	--	--	--	--
Orbaloid			--	18	35	53	70	35	18	35
ODR 370°F										
	Min		18	19.0	15.5	14.5	11.3	17.0	14	13
	Max		65	82.0	79.5	81.0	79.0	85.0	82	63
	T4		.9	1.0	1.1	1.3	1.2	.9	1.0	1.0
	Tc		1.8	2.0	2.0	2.2	2.2	1.8	1.9	1.9
ML1+4/212°F			67	--	--	--	--	--	52	50
MS at 250°F										
	T4		12	16.0	19.4	25.2	29.0	13.5	15	13
	Min		34	32.6	22.8	17.3	12.8	35.0	23	22
MODULUS - 100 (PSI)	-30°F	30	625	425	--	--	--	615	390	400
	72°F	10	485	360	340	250	210	395	350	355
		20	520	390	360	265	205	450	360	395
		30	570	410	340	240	200	500	375	385
	250°F	30	335	240	--	--	--	260	245	255
MODULUS - 200 (PSI)	-30°F	30	1415	1010	--	--	--	1070	970	940
	72°F	10	1325	895	680	465	350	875	840	840
		20	1390	955	760	465	340	970	900	910
		30	1365	990	690	450	340	940	940	900
	250°F	30	575	410	--	--	--	420	530	465
MODULUS - 300 (PSI)	-30°F	30	2125	1875	--	--	--	1825	1830	1625
	72°F	10	2300	1690	1290	840	540	1570	1550	1520
		20	2400	1785	1585	855	535	1710	1660	1615
		30	2350	1790	1315	815	540	1700	1765	1580
	250°F	30	1015	650	--	--	--	660	680	770
MODULUS - 400 (PSI)	-30°F	30	--	--	--	--	--	--	--	--
	72°F	10	3025	2592	2250	1550	1050	2390	2340	2280
		20	3360	2725	2315	1540	1000	2510	2490	2370
		30	3180	2792	2060	1440	1005	2520	2635	2285
	250°F	30	1505	960	--	--	--	970	1000	1120
TENSILE (PSI)	-30°F	30	2600	2600	--	--	--	2550	2185	2050
	72°F	10	3775	3450	3010	2955	2540	3000	3120	3050
		20	3665	3438	3240	2715	2570	2980	3350	3000
		30	3835	3358	3140	2590	2405	2880	3530	2925
	250°F	30	2300	1750	--	--	--	1620	1920	1890

TABLE 12: CONT'D.

COMPOUND	CURE*	F-12	F-13	F-14	F-15	F-16	F-17	F-18	F-19
ELONGATION									
-30°F	30	360	370	--	--	--	350	360	350
72°F	10	520	493	455	535	535	480	460	500
	20	680	475	495	525	560	465	515	490
	30	500	462	510	533	545	445	515	490
250°F	30	560	640	--	--	--	590	680	620
SHORE A									
72°F	10	65	64	60	53	67	67	64	66
	20	70	65	60	53	48	68	65	67
	30	70	65	60	53	48	68	65	68
TEAR DIE B									
(LB/IN) -30°F	30	--	--	--	--	--	--	--	--
72°F	10	745	802	593	287	174	542	633	531
	20	781	764	566	221	128	560	623	461
	30	777	731	503	250	120	560	667	521
250°F	30	417	311	--	--	--	259	323	235
AGED 70 HRS/158 F									
MODULUS - 100		650	480	--	--	--	540	490	450
MODULUS - 200		1775	1110	--	--	--	1120	1200	1035
MODULUS - 300		3075	2040	--	--	--	2000	2090	1840
MODULUS - 400		3725	2980	--	--	--	2950	3000	2670
TENSILE-(PSI)		3875	3350	--	--	--	2950	3575	3000
ELONGATION		420	440	--	--	--	400	480	440
SHORE A		71	70	--	--	--	71	66	69
DEMATTIA FLEX									
(kc/0.1 in)	30	47	19	--	--	--	12	14	7
BFG FLEXOMETER									
TEMP (3 MIN)		86	58	--	--	--	71	65	32
TEMP (6 MIN)		108	66	--	--	--	81	38	95
MIN TO BLOW		6	27	--	--	--	21	18	12
PICO ABRADER, %		153	88	--	--	--	68	79	61
COMPRESSION SET, %									
-30°F		72	70	--	--	--	81	73	71
72°F		11	10	--	--	--	9	10	11
212°F		46	54	--	--	--	53	43	48
CHIPPING & CHUNKING								7	

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)

rubber. Linear decreases in hardness, modulus, tensile strength and tear resistance were observed with the level of Orbaloid replacement of carbon black. In natural rubber (as with guayule) partial replacement of carbon black with orbaloid decreased the abrasion and cut growth resistance with some gain in blowout resistance. Similarly, additional orbaloid increased the modulus, but further reduced the abrasion resistance, flex cut growth resistance and the advantage in blowout resistance.

Compared to current SBR tank track pad vulcanizates, which are shown in Table 17, the partial Orbaloid replacement of carbon black in guayule and natural formulae produced lower abrasion resistance, intermediate blowout resistance but better tear and flex cut growth resistance.

D. Guayule and Natural Rubber Reinforced with Nucap 200

Nucap is kaolin with a silane coupling agent which improves the reinforcement of rubber by the kaolin. This material is frequently utilized in the rubber industry.

Nucap as a partial replacement for the carbon black in guayule compounds reduced the abrasion resistance, modulus, hot tear strength and flex cut growth resistance as shown in Table 13.

Additional Nucap increased the hardness to the level of the all carbon black recipe, but did not increase the modulus and further reduced the abrasion resistance, tear strength, flex cut growth resistance and time to blowout.

The properties of the natural rubber vulcanizates evidenced the same trends observed in the studies employing guayule rubber.

Compared to the current SBR tank track pad compounds shown in Table 17, the vulcanizates containing guayule and Nucap evidenced lower abrasion resistance and time to blowout, but better flex cut growth resistance and tear resistance at low and elevated temperatures.

TABLE 13: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH NUCAP 200

COMPOUND		F-2	F-20	F-21	F-27	F-23	F-24
Guayule		100	--	--	--	100	100
Natural		--	100	100	100	--	--
N234 Black		62	47	47	--	47	47
N110 Black		--	--	--	47	--	--
Nucap 200		--	22	45	22	22	45
ODR 370°F							
Min		18	19.8	22.0	18.0	17	17
Max		65	74.5	70.3	69.2	58	50
T4		.9	1.3	1.0	1.1	1.0	1.0
Tc		1.8	2.0	2.0	2.1	1.8	1.9
ML1+4/212°F		67	--	--	--	60	68
MS at 250°F							
T4		12	15.0	11.3	18.5	14	10
Min		34	33.3	43.4	30.5	26	34
	CURE*						
MODULUS - 100							
(PSI) -30°F	30	625	710	--	--	540	730
72°F	10	485	390	605	395	400	540
	20	520	450	650	435	460	570
	30	570	430	645	400	440	535
250°F	30	335	210	--	--	265	300
MODULUS - 200							
(PSI) -30°F	30	1415	1425	--	--	1215	1390
72°F	10	1325	950	1340	955	970	1165
	20	1390	1085	1360	980	1080	1170
	30	1365	1045	1380	960	1040	1150
250°F	30	575	425	--	--	445	575
MODULUS - 300							
(PSI) -30°F	30	2125	2180	--	--	1925	2065
72°F	10	2300	1710	2060	1640	1750	1900
	20	2400	1920	2140	1725	1880	1900
	30	2350	1905	2175	1690	1830	1890
250°F	30	1015	660	--	--	660	825
MODULUS - 400							
(PSI) -30°F	30	--	--	--	--	2710	--
72°F	10	3025	2585	2805	2500	2520	2650
	20	3360	2840	2855	2530	2705	2640
	30	3180	2785	2910	2500	2640	2630
250°F	30	1505	940	--	--	950	1160
TENSILE							
(PSI) -30°F	30	2600	2840	--	--	2800	2430
72°F	10	3775	3845	3275	3715	3775	3300
	20	3665	3915	3210	3700	3750	3400
	30	3835	3560	3065	3610	3850	3400
250°F	30	2300	1840	--	--	2090	2200

TABLE 13: CONT'D.

COMPOUND		F-2	F-20	F-21	F-27	F-23	F-24
ELONGATION, %							
-30°F	30	360	390	--	--	410	360
72°F	10	520	555	565	530	560	500
	20	480	525	555	540	540	520
	30	500	505	530	540	560	520
250°F	30	560	730	--	--	760	750
SHORE A							
72°F	10	65	58	65	60	65	67
	20	70	60	66	62	66	69
	30	70	60	67	61	66	69
TEAR DIE B							
(LB/IN) -30°F	30	--	--	--	--	--	--
72°F	10	745	736	342	972	732	423
	20	781	815	232	635	693	400
	30	777	749	233	670	641	402
250°F	30	417	410	--	--	364	367
AGED 70 HRS/158°F							
MODULUS - 100		650	670	--	--	570	760
MODULUS - 200		1775	1480	--	--	1325	1580
MODULUS - 300		3075	2440	--	--	2025	2430
MODULUS - 400		3725	3350	--	--	3040	3250
TENSILE-(PSI)		3875	3800	--	--	3850	3500
ELONGATION		420	460	--	--	515	450
SHORE A		71	68	--	--	69	72
DEMATTIA FLEX							
(kc/0.1 in)		47	33	--	--	28	21
BFG FLEXOMETER							
TEMP (3 MIN)		86	74	--	--	80	104
TEMP (6 MIN)		108	96	--	--	97	6
MIN TO BLOW		6	12	--	--	9	6
PICO ABRADER, %		153	103	--	--	80	67
COMPRESSION SET, %							
-30°F		72	78	--	--	86	92
72°F		11	9	--	--	11	16
212°F		46	55	--	--	46	55

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)

E. Guayule and Natural Rubber Reinforced with Austin Black

Table 14 shows the results produced by total or partial replacement of carbon black with Austin Black in the Guayule and Natural Rubber compounds. Abrasion resistance, modulus at low, ambient and elevated temperatures, as well as tear strength in guayule rubber vulcanizates were reduced. However, time to blowout was significantly improved. Partial replacement of N234 carbon black by Austin Black also produced a significant reduction in flex cut growth resistance.

Additional Austin Black produced a hardness and modulus increase, but further reduced the abrasion resistance, adversely affected by the low temperature compression set, and reduced the improvement in blowout resistance.

Compared to current SBR tank track pads, shown in Table 17 the partial replacement of carbon black with Austin Black produced vulcanizates with significantly poorer abrasion resistance, but significantly better ambient tear strength and resistance to flex cut growth.

F. Guayule Natural Rubber Reinforced with Santoweb D, DX - HiSil

Santoweb D is short celullose fibers treated with a bonding agent; Santoweb DX is untreated (15). These materials have been shown to improve the chipping resistance of an SBR vulcanizate containing carbon black and HiSil. In our experiments,50% blends of Santoweb D or DX and HiSil Silica were used and the results are given in Tables 15 and 16.

In Table 15 it is shown that the replacement of 25 to 50% of N234 carbon black with the above materials in a guayule vulcanizate produced a significant adverse effect on the abrasion resistance, tensile strength, hot tear resistance, flex cut growth, heat generation and low temperature compression set.

TABLE T4: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH AUSTIN BLACK

COMPOUND		CURE*	F-2	F-25	F-26	F-27	F-28	F-29	F-30	F-31	F-32
Guayule			100	--	--	--	--	--	100	100	100
Natural			--	100	100	100	100	100	--	--	--
N234 Black			62	47	31	16	--	47	47	47	--
N110 Black			--	--	--	--	--	--	--	--	--
Austin Black			--	12	23	35	46	23	12	23	46
ODR 370°F	Min		18	17.0	16.0	14.5	13.0	17.0	16	18	14
	Max		65	80.5	79.0	77.3	76.0	82.0	68	69	59
	T4		.9	1.0	1.1	1.2	1.4	1.0	.9	1.0	1.4
	Tc		1.8	2.1	2.1	2.3	2.6	2.1	1.8	1.9	2.6
ML1+4/212°F			67	--	--	--	--	--	62	65	42
MS at 250°F	T4		12	--	--	--	--	--	15	16	47
	Min		34	--	--	--	--	--	27	31	18
MODULUS - 100 (PSI)	-30°F	30	625	495	--	--	--	--	540	600	345
	72°F	10	485	390	315	255	165	440	390	450	225
		20	520	415	345	305	220	480	450	525	240
		30	570	440	365	305	220	455	425	515	220
	250°F	30	335	230	--	--	--	--	320	320	185
MODULUS - 200 (PSI)	-30°F	30	1415	1115	--	--	--	--	1080	1150	580
	72°F	10	1325	900	690	555	380	940	925	965	410
		20	1390	960	750	645	480	1035	1050	1070	490
		30	1365	1010	765	600	440	1000	1010	1075	475
	250°F	30	575	420	--	--	--	--	455	520	250
MODULUS - 300 (PSI)	-30°F	30	2125	1945	--	--	--	--	1875	1800	930
	72°F	10	2300	1610	1190	945	660	1660	1660	1680	710
		20	2400	1760	1335	1065	800	1800	1840	1800	830
		30	2350	1830	1345	1010	775	1725	1820	1775	805
	250°F	30	1015	650	--	--	--	--	710	755	290
MODULUS - 400 (PSI)	-30°F	30	--	--	--	--	--	--	2800	--	1415
	72°F	10	3025	2470	1940	1575	1100	2500	2460	2500	1250
		20	3360	2600	2100	1750	1325	2650	2700	2550	1420
		30	3180	2660	2110	1610	1300	2450	2700	2560	1355
	250°F	30	1505	950	--	--	--	--	1025	1015	335
TENSILE (PSI)	-30°F	30	2600	2610	--	--	--	--	2800	2165	2600
	72°F	10	3775	3550	3110	2880	2850	3000	3630	3250	2450
		20	3665	3300	3125	2770	2300	3010	3650	3300	2650
		30	3835	3410	3025	2710	2380	2775	3680	3200	2540
	250°F	30	2300	1750	--	--	--	--	1420	2000	600

TABLE I4: CONT'D.

COMPOUND		F-2	F-25	F-26	F-27	F-28	F-29	F-30	F-31	F-32
ELONGATION, %										
-30°F	30	360	380	--	--	--	--	400	465	600
72°F	10	520	443	435	450	575	475	555	500	540
	20	480	486	425	490	520	455	520	500	545
	30	500	495	400	512	525	455	520	495	545
250°F	30	560	650	--	--	--	--	510	760	580
SHORE A										
72°F	10	65	56	60	54	49	66	64	68	46
	20	70	65	60	55	52	67	66	69	50
	30	70	65	60	55	52	67	66	69	50
TEAR DIE B										
(LB/IN) -30°F	30	--	--	--	--	--	--	--	--	--
72°F	10	745	688	472	393	178	545	704	692	235
	20	781	722	551	384	195	570	668	652	248
	30	777	763	575	302	210	493	718	605	164
250°F	30	417	425	--	--	--	--	278	309	118
AGED 70 HRS/158°F										
MODULUS - 100		650	530	--	--	--	--	500	625	265
MODULUS - 200		1775	1225	--	--	--	--	1150	1310	540
MODULUS - 300		3075	2140	--	--	--	--	2000	2150	905
MODULUS - 400		3725	3050	--	--	--	--	2900	2975	1550
TENSILE (PSI)		3875	3575	--	--	--	--	3700	3350	2590
ELONGATION		420	460	--	--	--	--	500	450	515
SHORE A		71	70	--	--	--	--	69	70	51
DEMATTIA FLEX										
(kc/0.1 in)		47	21	--	--	--	--	11	8	14
BFG FLEXOMETER										
TEMP (3 MIN)		86	56	--	--	--	--	60	68	23
TEMP (6 MIN)		108	63	--	--	--	--	67	82	29
MIN TO BLOW		6	27	--	--	--	--	27	15	30+
PICO ABRADER		153	100	--	--	--	--	92	76	24
COMPRESSION SET, %										
-30°F		72	73	--	--	--	--	77	84	63
72°F		11	10	--	--	--	--	10	3	3
250°F		46	52	--	--	--	--	41	41	37
CHIPPING & CHUNKING		4						2		

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)

TABLE 15: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH SANTOWEB D/HISIL

COMPOUND		F-2	F-33	F-34	F-35	F-36	F-37
Guayule		100	--	--	100	100	100
Natural		--	100	100	--	--	--
N234 Black		62	47	31	47	31	--
N110 Black		--	--	--	--	--	--
50/50 Santoweb/Hisil		--	14	27	14	27	54
ODR 370°F							
Min		18	21.0	19.2	18	21	12
Max		65	73.0	58.2	58	51	41
T4		.9	.9	1.0	1.0	1.1	1.8
Tc		1.8	1.9	2.0	1.9	2.0	2.9
ML1+4/212°F		67	--	--	65	53	42
MS at 250°F							
T4		12	13.0	13.3	16	19	70
Min		34	34.0	28.0	28	26	17
	CURE*						
MODULUS - 100 (PSI) -30°F	30	625	565	--	600	535	563
72°F	10	485	325	305	440	--	--
	20	520	350	315	450	465	570
	30	570	350	310	480	460	280
250°F	30	335	220	--	305	250	245
MODULUS - 200 (PSI) -30°F	30	1415	1220	--	1150	810	767
72°F	10	1325	800	585	900	740	--
	20	1390	825	600	920	740	760
	30	1365	900	580	990	730	400
250°F	30		335	--	460	320	250
MODULUS - 300 (PSI) -30°F	30	2125	1825	--	1775	1545	--
72°F	10	2300	1550	1050	1600	1340	--
	20	2400	1575	1100	1650	1260	1025
	30	2350	1660	1030	1675	1230	580
250°F	30	1015	540	--	740	430	260
MODULUS - 400 (PSI) -30°F	30	--	--	--	--	--	--
72°F	10	3025	2330	1700	2340	1825	--
	20	3360	2380	1750	2400	1825	1200
	30	3180	2500	1700	2430	1850	800
250°F	30	1505	820	--	1075	550	270
TENSILE (PSI) -30°F	30	2600	2460	--	2300	1930	1125
72°F	10	3775	3000	2475	3050	2275	--
	20	3665	3000	2325	3100	2440	1200
	30	3835	3075	2350	3075	2215	1240
250°F	30	2300	1565	--	1850	1280	1490

TABLE 15: CONT'D.

COMPOUND		F-2	F-33	F-34	F-35	F-36	F-37
ELONGATION, %							
-30°F	30	360	365	--	370	355	280
72°F	10	520	480	510	500	480	--
	20	480	475	480	505	490	400
	30	500	475	500	495	480	540
250°F	30	560	620	--	610	760	--
SHORE A							
72°F	10	65	64	60	65	65	--
	20	70	65	60	68	68	68
	30	70	65	60	68	68	68
TEAR DIE B							
(LB/IN) -30°F	30	--	--	--	--	--	--
72°F	10	745	705	757	606	302	--
	20	781	613	798	709	366	179
	30	777	643	696	612	265	173
250°F	30	417	384	--	254	219	124
AGED 70 HRS/158°F							
MODULUS - 100		650	535	--	515	545	350
MODULUS - 200		1775	1210	--	1070	870	515
MODULUS - 300		3075	2160	--	2100	1450	750
MODULUS - 400		3725	3010	--	2925	2155	1225
TENSILE (PSI)		3875	3280	--	3200	2440	1550
ELONGATION		420	425	--	440	440	495
SHORE A		71	69	--	70	66	74
DEMATTIA FLEX (kc/0.1 in)		47	22	--	17	7	2
BFG FLEXOMETER							
TEMP (3 MIN)		86	85	--	92	100	100
TEMP (6 MIN)		108	108	--	116	--	--
MIN TO BLOW		6	12	--	9	3	3
PICO ABRADER		153	100	--	76	41	37
COMPRESSION SET, %							
-30°F		72	84	--	87	79	87
72°F		11	8	--	11	12	12
250°F		46	54	--	51	52	73
CHIPPING & CHUNKING		4			4		

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)

TABLE 16: PROPERTIES MEASURED FOR THE GUAYULE AND NATURAL RUBBER SYSTEMS REINFORCED WITH SANTOWEB DX/HISIL

COMPOUND	MINUTES CURE*	F-1	F-2	F-38	F-39	F-40
Guayule		--	100	--	--	100
Natural		100	--	100	100	--
N234 Black		62	62	47	31	31
N110 Black		--	--	--	--	--
50/50 Santoweb DX/Hisil		--	--	14	27	27
ODR 370°F						
Min		27.5	18	17.5	14.8	19
Max		89.3	65	70.0	56.0	58
T4		.9	.9	.9	1.0	1.0
Tc		1.9	1.8	1.9	2.0	1.9
ML1+4/212°F		--	67	--	--	69
MS at 250°F						
T4		10.8	12	13.0	17.0	16
Min		50.0	34	29.5	21.0	31
MODULUS - 100 (PSI) -30°F	30	720	625	595	--	700
72°F	10	485	450	310	250	440
	20	520	475	335	295	455
	30	570	495	350	285	445
250°F	30	335	295	225	--	255
MODULUS - 200 (PSI) -30°F	30	1580	1415	1215	--	1315
72°F	10	1410	1325	760	495	925
	20	1510	1390	780	525	945
	30	1550	1365	850	500	925
250°F	30	660	575	340	--	395
MODULUS - 300 (PSI) -30°F	30	2250	2125	1900	--	1980
72°F	10	2420	2300	1440	860	1650
	20	2630	2400	1525	970	1700
	30	2660	2350	1620	870	1660
250°F	30	1180	1015	550	--	605
MODULUS - 400 (PSI) -30°F	30	--	--	--	--	--
72°F	10	3250	3025	2250	1450	2415
	20	3500	3360	2300	1600	2400
	30	3550	3180	2450	1450	2350
250°F	30	1710	1505	830	--	850
TENSILE (PSI) -30°F	30	2670	2600	2450	--	2170
72°F	10	3938	3775	3040	2440	3150
	20	4017	3665	3100	2495	3200
	30	4038	3835	3050	2475	3125
250°F	30	2410	2300	1600	--	1630

TABLE 16: CONT'D.

COMPOUND		F-1	F-2	F-38	F-39	F-40
ELONGATION, %						
-30°F	30	370	360	365	--	335
72°F	10	490	520	505	530	505
	20	467	480	495	515	500
	30	465	500	490	510	505
250°F	30	515	560	640	--	675
SHORE A						
72°F	10	68	65	60	59	67
	20	69	70	65	60	68
	30	69	70	65	60	68
TEAR DIE B						
(LB/IN) -30°F	30	--	--	--	--	--
72°F	10	921	745	717	382	649
	20	729	781	616	424	636
	30	669	777	682	289	682
250°F	30	596	417	387	--	258
AGED 70 HRS/158°F						
MODULUS - 100		700	650	525	--	550
MODULUS - 200		1880	1775	1200	--	1085
MODULUS - 300		3000	3075	2125	--	1920
MODULUS - 400		3850	3725	3025	--	2730
TENSILE (PSI)		3850	3875	3375	--	3025
ELONGATION		400	420	440	--	450
SHORE A		73	71	69	--	72
DEMATTIA FLEX (kc/0.1 in)		41	47	25	--	24
BFG FLEXOMETER						
TEMP (3 MIN)		68	86	84	--	98
TEMP (6 MIN)		89	108	122	--	124
MIN TO BLOW		7	6	6	--	6
PICO ABRADER, %		177	153	98	--	74
COMPRESSION SET, %						
-30°F		76	72	81	--	84
72°F		10	11	10	--	12
212°F		47	46	58	--	54

* CURE TIME IN MINUTES AT 298°F
(30 Minutes where not indicated)

At the 50% replacement level in blends with guayule rubber, Santoweb D produced a severe adverse effect on tensile strength, tear resistance, and flex cut growth. However, at the 25% replacement level these properties were equal or better than the same properties for a current SBR tank track pad.

The preliminary studies utilizing natural rubber vulcanizates evidenced effects paralleling the above results with guayule vulcanizates.

Table 16 shows the behavior of the Santoweb DX blends. The results are similar in nature to those observed for the Santoweb D blends. Abrasion resistance and tear are better in this system, but the material runs better in the BFG Flexometer. The Santoweb DX-carbon black system in general seems to produce adverse results at the 25% volume level.

G. Blends of Guayule Rubber with Other Polymers

It has been shown (8) that a polymer Tri-blend consisting of natural rubber, polybutadiene (BR), and SBR (in the ratio 60-28-12) yields improved abrasion and tear resistance compared to all SBR compounds of the type currently used in tank track pads. Table 17 compares guayule rubber to natural rubber in the Tri-blend compound. Also shown are data for compounds with various polymer ratios.

In the Tri-blend system containing natural and guayule rubber the system seems to produce slightly better hot tear resistance, reduced abrasion resistance and slightly lower modulus. The flex life was also somewhat improved while other properties were comparable to the guayule and natural rubber compounds.

The Tri-blends produced little improvement in time to blowout compared to all-guayule rubber or all-natural rubber compounds. Increased curatives in an all-guayule compound G-43 also failed to significantly improve the time to blowout, even though the heat build-up was improved.

TABLE 17: PROPERTIES MEASURED FOR THE POLYMER BLENDS REINFORCED WITH VARIOUS FILLERS

COMPOUND NO.	MINUTES CURE	E-41	E-42	G-43	H-44	H-45	G-46	G-47	SP-1	SP-2
Guayule		--	60	100	90	90	90	90	--	--
Natural		60	--	--	--	--	--	--	--	--
BR		12	12	--	10	--	10	10	--	--
SBR		23	28	--	--	10	--	--	--	--
N234		62	62	62	62	62	47	47	--	--
Austin Black		--	--	--	--	--	12	--	--	--
Santoweb/HiSil		--	--	--	--	--	--	14	--	--
ODR 370°F										
Min		20	21	29	26	24	17	16	11	10
Max		91	76	73	84	80	65	61	98	73
T4		1.1	1.1	0.9	1.0	0.9	1.2	1.2	1.6	1.5
Tc		1.9	1.8	1.8	1.8	1.8	2.2	2.1	2.8	2.6
ML1+4/212°F		60	62	82	76	74	65	60	50	48
MS at 250°F										
T4		17	16	10	12	10	26	23	48	31
Min		33	32	44	42	38	31	27	22	22
MODULUS - 100 (PSI) -30°F	30	550	520	500	650	67-	415	600	1400	1320
72°F	10	430	390	395	450	465	320	260	640	370
	20	475	410	460	560	495	365	350	660	390
	30	410	440	450	510	520	390	350	600	380
250°F	30	345	360	340	375	380	235	255	440	315
MODULUS - 200 (PSI) -30°F	30	1165	1065	1365	1285	1455	975	1225	--	1690
72°F	10	1100	980	1150	1275	1300	750	790	1650	890
	20	1260	1080	1340	1520	1420	875	765	1725	925
	30	1095	1120	1310	1375	1475	910	860	1600	900
250°F	30	780	790	730	755	820	435	435	995	775

TABLE 17: CONT'D.

COMPOUND NO.	MINUTES CURE	E-41	E-42	G-43	H-44	H-45	G-46	G-47	SP-1	SP-2
MODULUS - 300 (PSI)										
-30°F	30	1740	1665	2015	2160	2460	1675	1895	--	--
72° F	10	1940	1750	2110	2260	2310	1345	1435	2650	1525
	20	2180	1950	2330	2550	2390	1575	1445	2775	1600
	30	1930	1940	2320	2360	2490	1630	1570	2620	1525
250°F	30	1125	1150	1190	1200	1325	675	735	--	1185
MODULUS - 400 (PSI)										
-30°F	30	--	--	--	--	--	2395	--	--	--
72° F	10	2700	2510	2915	3100	3090	2080	2175	3300	2120
	20	2975	2710	3150	3375	3275	2350	2195	--	2225
	30	2680	2735	3125	3160	3320	2390	2345	--	2175
250°F	30	--	--	1550	1750	1975	--	1060	--	--
TENSILE (PSI)										
-30°F	30	2200	2080	2400	3000	2600	2500	2260	2625	1980
72° F	10	3400	3285	3760	3765	3740	3640	3045	3300	2940
	20	3385	3300	3900	3860	3725	3625	2960	3110	3025
	30	3325	3290	3755	3770	3800	3600	3165	3100	2960
250°F	30	1400	1420	2830	2550	2680	1535	1060	1350	1510
ELONGATION, %										
-30°F	30	350	390	390	360	340	450	375	170	220
72° F	10	505	535	535	505	500	610	535	400	550
	20	460	490	505	485	470	570	510	340	555
	30	495	480	485	485	470	560	520	370	540
250°F	30	340	390	580	510	530	350	400	265	390
SHORE A										
72°F	10	71	68	70	70	70	64	65	75	72
	20	72	70	72	72	72	65	65	76	72
	30	72	70	72	72	72	65	65	76	72

TABLE 17: CONT'D.

COMPOUND NO.		E-41	E-42	G-43	H-44	H-45	G-46	G-47	SP-1	SP-2
TEAR, DIE B										
(LB/IN) 72°F	10	708	666	1018	894	861	741	679	359	491
	20	729	654	872	836	770	725	750	299	532
	30	737	702	686	721	752	616	668	356	530
250°F	30	349	418	314	305	325	267	193	135	279
AGED 70 HRS/158°F										
MODULUS - 100	30	550	540	560	620	550	490	520	750	480
MODULUS - 200	30	1350	1330	1610	1670	1820	1100	1130	1900	1110
MODULUS - 300	30	2320	2230	2715	2780	2885	1950	1940	2900	1800
MODULUS - 400	30	3070	3000	3565	3650	3700	2850	2510	--	2450
TENSILE (PSI)	30	3350	3330	3940	3960	3810	3570	3290	3140	3070
ELONGATION, (%)	30	440	455	460	440	420	485	470	335	530
SHORE A	30	76	75	75	76	76	70	71	77	74
DEMATTIA FLEX (kc/0.1 in)		3	5	37	32	32	32	38	3	5
BFG FLEXOMETER										
TEMP (3 MIN)		61	67	52	61	61	58	65	61	77
TEMP (6 MIN)		85	85	85	83	83	68	90	80	115
MINUTES TO BLOW		8	7	7	8	8	23	7	30+	10
PICO ABRADER		162	151	134	159	156	104	97	122	116
COMPRESSION SET, %										
22 HRS/-30°F		57	62	83	68	78	69	76	79	81
72° F		11	10	13	13	14	9	13	15	24
212° F		59	43	44	40	41	42	57	27	34
CHIPPING & CHUNKING		2	9	2	5	5			0	0

* Cure Temperature 298°F (SP-1 and SP-2 Cured at 320 F)

Except for a loss in tear resistance and processing safety, all other physical properties were comparable.

Guayule rubber blends with either polybutadiene rubber or SBR (H-44, H-45) have not evidenced significant improvement in time to blowout. Note that curative levels were increased to compensate for the differences in cure rates of the polymers. However, a relatively good hot tear was retained.

The time to blowout was significantly improved by a filler blend of Austin Black and N234 carbon black in the polymer blend of guayule with polybutadiene. Santoweb/HiSil system, however, produced poor results in the same direction and also gave poorer hot-tear results than the Austin Black system. It becomes obvious from the results shown on Table 17 that the blends gave better flex life than the control SBR compounds SP-1 and SP-2, and in most cases their performance is superior.

H. Optimization of Guayule - Filler Systems

Table 18 gives the main properties of selected guayule/filler systems. These systems are compared with standard SBR compounds used in today's tank track pads. It becomes obvious from the results that the physical properties of the guayule/filler systems are better or equal to those of the SBR currently used compounds. Tear and heat generation of the SBR compounds are comparable to those of the guayule/filler systems while the flex life is extremely poor. The SBR compounds give better abrasion resistance than the optimized guayule-filler blends. However, the guayule system itself has an abrasion resistance which is superior to the blends and also to the SBR compounds.

A comparison of the chipping and chunking test results, aged for 3 days at 100° C. (212° F.) shows that the SBR compounds currently used in the tank track pads were unaffected by the testing technique, and they are first in performance with respect to both chipping and chunking.

TABLE 18: OPTIMIZED PROPERTIES OF GUAYULE - FILLER SYSTEMS

COMPOUND NO.	F-1	F-2	F-4	F-12	F-18	F-23	F-30	F-35	F-40	SP-1	SP-2
TENSILE (PSI)											
-30°F	2670	2600	2300	2600	2185	2800	2800	2300	2170	2625	1890
72°F	4038	3835	3475	3550	3530	3850	3680	3075	3125	3100	2960
250°F	2410	2300	1750	1860	1920	2090	1420	1850	1630	1350	1510
ELONGATION, %											
-30°F	370	360	385	400	360	410	400	370	335	170	220
72°F	465	500	530	510	515	560	520	495	505	370	540
250°F	515	560	660	600	680	760	510	610	675	265	390
TEAR, DIE B (LB/IN)											
72°F	669	777	742	745	677	641	718	612	82	356	530
250°F	598	417	360	307	323	364	278	254	258	135	279
GOODRICH FLEX											
TEMP (3 MIN)	68	86	68	72	65	80	60	92	98	61	77
TEMP (6 MIN)	89	108	86	83	88	97	67	116	124	80	115
MIN TO BLOW	7	6	15	15	18	9	27	9	6	30+	10
DEMATTIA FLEX (kc/0.1 in)	41	47	14	22	14	28	11	17	24	3	5
PICO ABRADER	177	153	85	69	79	80	92	76	74	122	116
CHIPPING & CHUNKING	2	4	9		7		2	4		0	0

The blends of all carbon black natural rubber and guayule rubber compounds were second in performance with respect to chipping. Blends of these systems with Austin Black and Santoweb D/HiSil fillers were also good in chipping performance.

Increased chipping with considerable chunking were observed for the blends with lignin and orbaloid fillers.

Chipping and chunking test results do not seem to correlate well with any of the properties shown in Table 18. Therefore, it is difficult to draw concrete conclusions on the usefulness of this test.

CONCLUSIONS

Reinforcement of guayule and natural rubber with non-oil dependent fillers; such as, lignin, Fillite, Orbaloid, Nucap, Austin Black, and Santoweb D and DX has shown to give poor physical properties when these fillers are used as 100% replacement of the carbon black in the compound.

Physical properties are significantly improved when the aforementioned fillers are used as a partial replacement of the carbon black. In such cases it is shown that most properties are equivalent or better than the properties of the SBR compounds currently used in the tank track pad compounds.

RECOMMENDATIONS

We found in this work that a combination of non-oil dependent fillers with carbon black can give a good reinforcement to the guayule or natural rubber compounds. This reinforcement is better or equal to the reinforcement of all carbon black SBR compounds currently used in tank track pad applications. Therefore, we recommend that tank track pads be built and tested in the laboratory and tank track field by using such guayule filler compounds.

Because of the difficulties encountered in finding guayule rubber in large quantities in the U.S.A., it is recommended that natural rubber be used in the place of guayule rubber for such experimentations. The performance of the natural rubber compounds used in this work was found to be comparable to that of the guayule rubber compounds. Therefore, replacement of guayule with natural rubber in such experiments would not change the results significantly.

Treatment of some of the fillers with bond promoting agents may improve the bonding of the rubber to the fillers and thus improve the reinforcing effect. We also recommend that some research be done in this area, especially with lignin, to see if such treatment can be helpful to track pad applications.

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16. **The standard Indonesian rubber SIR20 used for this study contained .20% dirt; 1% ash; .60% Nitrogen; .8% volatile matter; 30 for the initial value of Wallace Rapid Plasticity, and 40% plasticity retention index.**

Appendix A

Abbreviations and Explanations of Laboratory Testing Conditions

ODR	Oscilating Disc Rheometer Conditions: Temperature 370 F Oscillation 5 degrees Measurements: T4 Minutes to 4 point rise T90 Minutes to 90% cure Mn Minimum Torque (inch - lbs.) Mx Maximum Torque (inch - lbs.)
MS 250° F.	Mooney Viscometer - Small Rotor - 250 F Measurements: T4 Minutes to 4 point rise Mn Minimum Torque (Mooney units)
MS 212° F.	Mooney Viscometer - Large Rotor - 212 F Measurements: Torque after 4 minutes (Mooney units)
MOD - 100	Modulus (PSI) at 100% Elongation - Tested at Ambient Temperature.
MOD - 100 (-30° F.)	Modulus (PSI) at 100% Elongation - Tested at -30 F
MOD - 100 (250° F.)	Modulus (PSI) at 100% Elongation - Tested at 250 F
TENSILE	Tensile Strength (PSI) at break
ELONGATION	Elongation (%) at break
REBOUND	Rebound (%) - Bashore Resiliometer
C. SET (-30° F.)	Compression Set B (%) 22 hours at -30 F
PICO (WEAR)	Pico Abrasion Resistance (Knife Edge) - Reciprocal of Volume Loss Compared to Pico Standard as 100
DM FLEX (kc/0.1 in.)	Kilocycles to 0.1 inch cut growth (Pierced Test Pieces) - DeMattia Flexometer - 300 cycles per minute
BFG FLEX	Goodrich Flexometer (Dynamic Compression Fatigue) Conditions: Cycles Per Minute 1800 Load (PSI) 200 Temperature Oven (Of) 100 Measurements: Temp (3) Temp (Of) Increase After 3 Minutes Temp (6) Same After 6 Minutes Blow Minutes to Blow-Out

Appendix A - (Continued)

Chipping and Chunking

Rating	Description		
0	No effects	-	50 blows
1	Slight scuffing	-	50 blows
2	Moderate scuffing	-	50 blows
3	Slight cracking	-	50 blows
4	Moderate cracking	-	50 blows
5	Chipping	-	50 blows
6	Chipping	-	10 blows
7	Chipping	-	Less than 10 blows
8	Chunking	-	50 blows
9	Chunking	-	10 blows
10	Chunking	-	Less than 10 blows

Equipment: Polysar Corporation, Limited - Sarnia, Ontario, Canada

(Guillotine Type)

Weight: 5 lbs.

Edge: Blunt blade, radiused edge, 1/4 in. thick

Height: Dropped from 16 in. (40 cm)

Sample: Cured section simulating a truck tire tread.

Dimensions: 2 X 2 X 2 cm

Samples withstanding 50 blows without losing chips over 1 mm in diameter are considered chip resistant.